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CHARACTERIZATION OF COMPOSITE MATERIALS USING MILLIMETER-WAVE TECHNIQUES (PREPRINT)

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14. ABSTRACT
Millimeter-wave reflection and transmission measurements were performed on various composite materials in order to characterize the changes in the structural and optical properties after being subjected to thermal and mechanical degradation.

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Characterization of Composite Materials using Millimeter-wave Techniques

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***Abstract*: Millimeter-wave reflection and transmission measurements were performed on various composite materials in order to characterize the changes in the structural and optical properties after being subjected to thermal and mechanical degradation.**

I. Introduction And Background

Our motivation for this work has been centered on the development of NDE methods for dielectric materials to identify defects in either the fabrication of the components or through their extended use in high thermal and mechanically stressed environments. Two such harsh environments are aircraft turbine engines and high altitude air travel. Defects are often evident through delamination, coating separation, and other forms of material degradation. Our methods of identifying defect features in an imaging system are through quantifying changes in the reflectivity and transmissivity of the sample that are due to base changes in the structural and/or optical properties. While transmission evaluations can be easier to interpret, not all materials are highly transmissive to millimeter-wave (MMW) radiation nor do in-situ applications lend themselves to transmission techniques. This has motivated the development of reflectivity measurement techniques. Many reflecting systems have been discussed in recent years[1-3]. Since these past techniques, as well as ours, are all designed for particular materials, we have been focused on the identification of the nuances of the defect signatures that are dependent on specific MMW sensing systems. Materials we have studied thus far are an Oxide /Oxide and SiNC/SiC ceramic matrix composites (CMC).

II. Measurement Systems

Our past work has concentrated on reflection imaging and transmission interferometry [4]. Initial measurements on the several composite samples showed that they were very highly absorbing such that much of the data measured in transmission from 132 to 240 GHz had a very low signal-to-noise ratio. This led to the development of methods for measuring the spectra in reflection mode.

Our setup for measuring reflection was made with normal incidence with the source and detector coupled via a mylar beam splitter. In both the imaging system and the reflection system a collimated beam was focused on to the sample via an off axis parabolic mirror (f/1.5), which provided high spatial resolution (diffraction limited) and a short depth of focus. A measure of the 3-D spatial profile of the focused beam was completed by mapping the reflected intensity off of a XYZ positioned mirror. The raster scanned imaging system made use of a similar optical design. Coupon and reference sample images were acquired at specific frequencies in the 80-120 GHz range.

III. Results

Continuous wave systems are known for standing wave artifacts which are a result of the reflections between any normal surfaces in system, such as the sample and source/detector. This results in an etalon-line baseline that reduces the accuracy of the measurements. Each system made use of AM and FM modulation and careful consideration of the optical design to reduce the standing wave effects. The reflectance system frequency swept from 132 to 240 GHz at 1 kHz[4], of which only the range from 142 to 198 GHz had enough power to produce reliable reflection results. This region is associated with the 1/*e* point of the source diode manufacturer's power curve [5]. Reflection values measured outside this region varied widely.

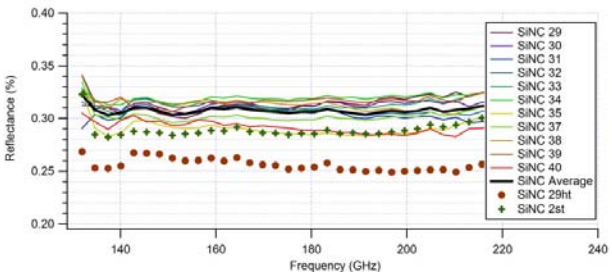

Figure 1. Reflection measurements of un-backed SiNC/SiC sample as compared against an aluminum reference. The continuous lines are baseline sample measurements. The two dotted lines are treated samples where one was mechanically stressed while the other was thermally stressed.

Another source of error was thickness and flatness variations in the sample. For our CMC coupons there were thickness and flatness issues with variances in thickness up to 0.25 mm and sample flatness varying as much as 0.36 mm over a 100 mm length. At 240 GHz the wavelength is 1.3 mm, so sample flatness would be a concern. We compensated for this by gripping the sample within 10 mm of the spot being measured. This constrained the coupon geometry to ensure a flat portion of the sample was measured normal to the MMW radiation. This sample holding method held the coupon front reflection surface at the same plane in relation to the focusing mirror, thus all thickness variations were on the rear surface. These variations in thickness accurately describe the etalon walk-off seen in Oxide/Oxide samples, shown in Fig. 2, as described by:

$$\Delta\phi = \frac{2n}{c}\Delta d \qquad \text{Eq. (1)}$$

where $\Delta\phi$ is the phase shift, n is the index of refraction, c is the speed of light, and Δd is the thickness difference between two spots.

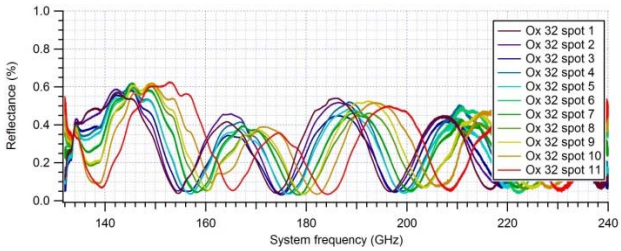

Figure 2. Reflection measurements of un-backed Oxide/Oxide sample as compared against an aluminum reference. The spots are associated with specific regions of the sample. The etalon walk-off is associated with nearly linear sample thickness variation from one end to the other.

The reflectance was modeled in terms of a thin film as:

$$R = R_t + R_e \cos\left(2\pi \frac{\nu}{\Delta\nu}\right) \qquad \text{Eq. (2)}$$

where R is the measured reflectance, R_t is the reflectance from the top surface, R_e is the reflectance associated with the etalon effect, and $\Delta\nu$ is the period of the etalon. Results from this model provided the surface reflectance and the index of refraction as shown in Table 1.

	Reflectance (%)	Index of Refraction	Thickness (mm)
Oxide/Oxide sample 32	0.304(12)	2.43(8)	2.77(7)
SiNC/SiC samples	0.307(2)	–	2.19(4)

Table 1. Summary of results from the reflectance system based on measurements over a frequency range of 140 to 200 GHz.

An image is shown in Fig. 3 of an aluminum reference and three SiNC/SiC samples: mechanically stressed, thermally stressed, and an unstressed baseline sample. From beam profile analyses, the beam waist is elliptical with approximate dimensions of 4 x 7 mm at 120 GHz. Our current method of analyzing image data is to take an average of the intensity measurements over one dimension of the image and divide it by the intensity of an aluminum reference. An example of an intensity line profile is shown below the image in Fig. 3. Mitigating the standing wave effects in the imaging system is more challenging since the sample could not be constrained to reduce the affects of the sample dimensional variation. Subtle interference bands can be seen across the sample. From carefully selected regions of the images, average intensities can be determined. Using this method, the unstressed SiNC/SiC samples have an average reflectance of 0.312(13). Further image analysis is on-going to determine if there is any significant difference between the unstressed and stressed samples. While there appears to be a significant difference in the image below, some of this difference is due to sample position on the imaging stage. Additional etalon fringes in the less absorbing Oxide/Oxide CMC samples further complicate the image analysis. Further work will include experimental modifications and etalon models to compensate and account for these standing wave effects.

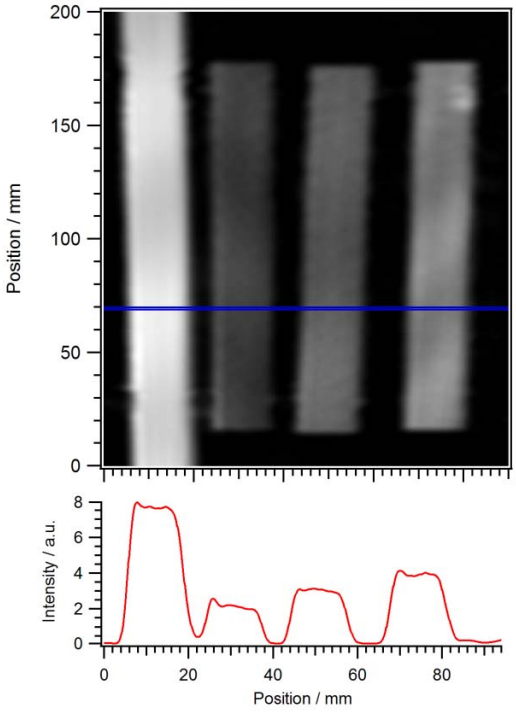

Figure 3. Image of an Aluminum reference and three SiNC/SiC samples (thermal, mechanical, unstressed) recorded at 92 GHz.

IV. CONCLUSION

We have measured the reflectance of the SiNC/SiC and Oxide/Oxide samples and index of refraction of the Oxide/Oxide samples using two different systems. Further work will determine if there are significant differences between thermally and/or mechanically degraded samples and untreated samples.

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